

# PATENT SPECIFICATION

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(72) Inventors SAKAE OGATA and GORO WAKABAYASHI



## (54) METHOD OF PREPARATION OF ACRYLIC POLYMER LATICES

(71) We, HIGH POLYMER CHEMICAL INDUSTRIES LIMITED, a Japanese body corporate of 25, 1-chome, Kushincho, Kita-ku, Osaka, Japan, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of preparing stable and thick acrylic polymer latices.

It is known that stable latices can readily be obtained when acrylic esters and/or methacrylic esters are emulsion polymerised along with vinyl acetate in an amount exceeding 25%, by weight, based on the total monomers, using as a protective colloid a water-soluble high-molecular-weight substance such as polyvinyl alcohol, tragacanth gum or hydroxyethyl cellulose. However, in those cases where the comonomer is not used at all or is used in a relatively small amount, a stable acrylic polymer latex, and particularly a stable acrylic polymer latex of high concentration, cannot be obtained when the protective colloid is present, since the emulsion breaks during the polymerisation reaction. While this shortcoming can be improved to a certain extent by the use of a surfactant instead of the protective colloid the properties of the acrylic polymer latex, such as film forming ability, miscibility with pigments and salts, tensile strength, water and solvent resistance, adhesiveness and film blocking resistance, are adversely affected.

We have now found that stable, concentrated, acrylic polymer latices which possess a good balance of properties can be prepared by the emulsion polymerisation of an acrylic and/or methacrylic ester, and optionally therewith a minor quantity of a comonomer, in the presence of a water-soluble high-molecular-weight substance as a protective colloid, using a method which comprises adding to the polymerisation system as an emulsion stabiliser 0.01—10 percent by weight, based on the total monomer, of allyl alcohol, an ester of allyl alcohol with an alkanolic acid, an allyl halide or a saturated aliphatic alcohol of 2—8 carbon atoms.

Acrylic esters and methacrylic esters which may be polymerised in accordance with the present invention include the methyl, ethyl, butyl and 2-ethylhexyl esters, as well as the glycidyl and hydroxyethyl esters.

Comonomers which can optionally be copolymerised, in minor amount, with the acrylic and/or methacrylic esters include;

1) One or more of acrylic acid, methacrylic acid and vinyl sulphonic acid, and their water-soluble salts in an amount up to 5% by weight based on the total monomers.

2) Maleic esters and/or fumaric esters, in an amount up to 30% by weight based on the total monomers.

3) Acrylonitrile and/or styrene, in an amount up to 30% by weight based on the total monomers.

4) Vinyl acetate and/or vinyl propionate, in an amount up to 25% by weight based on total monomers.

5 The protective colloid which is used in the method of the invention may be any natural, modified natural or synthetic water-soluble high-molecular-weight substance which is used in the conventional method of preparing acrylic polymer latices and it may be used in the customary manner and amounts. As 10 natural substances, mention may be made of tragacanth gum, gum arabic, starch, casein, gelatin and alginates, and, as modified natural substances, mention can be made of such as dextrin, hydroxyethyl cellulose, methyl 15 cellulose and carboxymethylcellulose. Suitable synthetic substances include polyacrylic acid and its salts, water-soluble acrylic acid copolymers and their salts, polyacrylamide, water-soluble acrylamide copolymers, water-soluble fumaric acid copolymers and their salts, poly- 20 methylolacrylamide, water-soluble methylolacrylamide copolymers, polyvinyl alcohol, partially esterified polyvinyl alcohol, 25 denatured polyvinyl alcohol and other water-soluble polyamides.

The polymerisation initiators which are conventionally used in emulsion polymerisation are used in the usual manner in the present 30 invention. For example, there can be used redox catalysts which combine an oxidising agent such as ammonium persulphate, potassium persulphate, hydrogen peroxide, t-butyl hydroperoxide and cumene hydroperoxide with a reducing agent such as acid sodium sulphite, 35 sodium thiosulphate, thiourea, ascorbic acid and ferrous sulphate in a combined amount of from 0.05—0.5% by weight based on the total emulsion. It is, of course, also possible to initiate the polymerisation reaction by irradiation by means of actinic radiation instead of 40 using chemical polymerisation initiators.

Examples of emulsion stabilisers which may be used in accordance with the invention 45 include allyl alcohol, allyl acetate, allyl chloride, allyl bromide, ethyl alcohol, propyl alcohol, butyl alcohol, octyl alcohol, ethylene glycol, propylene glycol, butanediol, 1,8-octanediol and methylisobutylcarbinol. While there are differences in the optimum quantities 45 of the various emulsion stabilisers, generally speaking, they should be used in an amount of 0.01 to 10% by weight, and preferably 0.05 to 5% by weight, based on the total monomers. 55 When the amount is less than 0.01% by weight, the desired emulsion stabilising effect is not demonstrated, whereas when the amount exceeds 10% by weight, the rate of polymerisation falls and the stability of the latex 60 is impaired rather than improved.

Various other substances may be added, if desired, to the polymerisation system, such as caustic alkalis as emulsion neutralisers, titanil sulphate to impart water resistance, a silicone

liquid as an anti-foamer, and a surfactant to reduce viscosity. 65

The emulsion polymerisation method of the invention is carried out in a conventional manner so that the monomer (or an aqueous dispersion of the monomer) can be added 70 dropwise to an aqueous solution of protective colloid; the emulsion stabiliser may be added to either the monomer or the aqueous protective colloid solution. Further, the addition may be made at once, continuously or inter- 75 mittently. A technique which is generally recommended in carrying out the emulsion polymerisation reaction is that consisting of dissolving the protective colloid (and, if neces- 80 sary, a small quantity of surfactant) in water, raising the temperature of this solution to the polymerisation initiating temperature, then adding the emulsion stabiliser to this solution, followed by addition of the polymerisation 85 initiator, and thereafter adding the monomer dropwise while controlling the reaction temperature. While the reaction temperature is not an essential factor of the present invention, a temperature of 0—60°C. is used when employing the redox catalysts and 60—80°C. 90 is used in the case of other conventional catalysts.

An acrylic polymer latex having excellent properties can be obtained by the method of the present invention. The latex can be used 95 with advantage in a wide variety of applications. It can thus be used as a textile treatment agent (as a binder for bonding cloth or in the flocking process or the manufacture of non-woven fabric, or as a printing paste or hand 100 imparting agent for cloth), as an adhesive (for plastic films or metallic foil), in paints, as a paper processing agent (clay coating agent or size) or as a sealing agent.

It is not clear why the acrylic polymer latex 105 produced by the method of the invention should possess improved properties but it is thought that graft polymerisation between the protective colloid and the acrylic and/or methacrylic ester is checked by the presence of the emulsion stabiliser. This would prevent 110 coagulation of the emulsion which would take place as a result of the bonding of the particles to each other.

The following Examples serve to illustrate 115 the invention. In the Examples percentages are by weight unless indicated otherwise.

#### EXAMPLE 1—4

A 5-necked 1-liter flask disposed in a hot water bath and equipped with a reflux con- 120 denser, a thermometer, a stirrer and a dropping funnel was charged with 250 grams of 4% aqueous polyvinyl alcohol solution (completely saponified product, degree of polymerization 1000), after which the emulsion stabilizer 125 indicated in Table I was added. After raising the temperature to 75°C. with stirring, 0.8

gram of potassium persulfate was added drop-  
wise, and at the same time the addition of 190  
grams of monomeric ethyl acrylate from the  
dropping funnel was begun. The dropping of  
the monomer was continued thereafter over a  
period of 2 hours  $\pm$  10 minutes at a tempera-  
ture as near constant as possible, the reaction  
temperature being kept at 80—85°C. After  
all of the monomer had been added, the tem-  
perature was held at 80—85°C. for another  
hour. The system was then cooled to below  
30°C. at which temperature the latex was  
removed from the flask.

When, by way of comparison, the foregoing  
operation was repeated but without using the  
emulsion stabilizer, disruption of the emul-  
sion took place during the polymerization.  
The latex obtained as described hereinbefore  
was applied to a glass sheet and air-dried.

TABLE I

|                         | Amount of<br>Polyvinyl<br>Alcohol<br>added (g) | Emulsion Stabilizer   |                        | Water<br>Resistance |                   | Resistance to Acetone     |                         | Resistance to<br>Trichloroethylene |                         |
|-------------------------|--|-----------------------|------------------------|---------------------|-------------------|---------------------------|-------------------------|------------------------------------|-------------------------|
|                         |  | Class                 | Amount<br>added<br>(g) | Swelling<br>(%)     | Solubility<br>(%) | Swelling<br>degree<br>(%) | Solubility<br>(%)       | Swelling<br>Degree<br>(%)          | Solubility<br>(%)       |
| Conventional<br>product | —  | —                     | —                      | 78                  | 0.4               | completely<br>dissolved   | completely<br>dissolved | completely<br>dissolved            | completely<br>dissolved |
| Example 1               | 10   | allyl acetate         | 10                     | 19                  | 0.3               | 48                        | 61.1                    | 320                                | 63.1                    |
| Example 2               | 10   | allyl alcohol         | 8                      | 15                  | 0.3               | 40                        | 37.4                    | 250                                | 58.0                    |
| Example 3               | 10   | sec. butyl<br>alcohol | 5                      | 20                  | 0.3               | 46                        | 62.0                    | 330                                | 62.5                    |
| Example 4               | 10   | 1,8-octane-<br>diol   | 15                     | 18                  | 0.3               | 47                        | 60.8                    | 320                                | 62.0                    |

Seven parts by weight of methylated trimethylol melamine was mixed with 100 parts by weight of the latex of Example 3, and this mixture was applied to a sheet glass and air-dried. After heating the resulting film for 5 minutes at 140°C., it was measured for its degree of swelling and solubility when dipped in perchloroethylene. A similar measurement was also made on a film obtained from a mixture of 100 parts by weight of the conventional latex and 7 parts by weight of methylated trimethylol melamine. As is apparent from Table II, the enhanced effect with respect to the resistance to solvents resulting from the addition of the melamine resin was much greater in the case of the latex of the invention than in the case where the conventional product was used.

TABLE II

## Resistance to Perchloroethylene

| Latex                | Swelling (%) | Solubility (%) |
|----------------------|--------------|----------------|
| Conventional product | 22.9         | 26.3           |
| Example 3            | 9.1          | 0              |

## EXAMPLE 5—6

As the protective colloid, a mixture of 250 grams of 5% aqueous gum arabic solution, 3 grams of hydroxyethyl cellulose and 0.1 gram of sodium lauryl sulfate was used, and a monomeric mixture consisting of 100 grams of n-butyl methacrylate, 3 grams of glycidyl methacrylate, 50 grams of 2-ethylhexyl acrylate, 4 grams of acrylic acid and 20 grams of acrylonitrile was emulsion polymerized using the apparatus employed in Examples 1—4 and operating as described therein. As the polymerization initiator, a redox catalyst consisting of 2 grams of ammonium persulfate and 3 grams of ascorbic acid was used, while as the emulsion stabilizer either 8 grams of allyl chloride (Example 5) or 12 grams of octyl alcohol (Example 6) was used.

When, by way of comparison, the foregoing

operation was repeated without using the emulsion stabilizer, disruption of the emulsion took place during the polymerization reaction.

When the latices obtained were used as adhesives to bond a cotton cloth to polyethylene terephthalate film, the peeling forces indicated in Table III were found to be necessary to separate the cloth from the film. The conventional product of Table III refers to the latex that is obtained from the foregoing monomeric mixture by means of the conventional emulsion polymerization method which uses a surfactant.

TABLE III

| Latex                | Peeling Force (g/cm) |
|----------------------|----------------------|
| Conventional product | 630                  |
| Example 5            | 1560                 |
| do 6                 | 1090                 |

## EXAMPLES 7—10

A monomeric mixture consisting of 130 grams of n-butyl acrylate, 30 grams of methyl methacrylate, 2 grams of 2-hydroxyethyl methacrylate, 20 grams of styrene, 6 grams of methylol acrylamide and 2 grams of methacrylic acid was emulsion polymerized using the apparatus employed in Examples 1—4 and operating as described therein, using as the protective colloid 250 grams of 4% aqueous solution of each of the various substances indicated in Table IV, and as the emulsion stabilizer, a mixture of 4 grams of allyl alcohol and 0.5 gram of n-propyl alcohol.

The latex obtained was applied to a glass sheet and air-dried, after which the resulting film was peeled and heated for 3 minutes at 150°C. The film obtained was dipped in perchloroethylene and measured for its degree of swelling and solubility. The results are shown in Table IV. That referred to as conventional product in Table IV is a latex prepared in accordance with the conventional method using a surfactant but without using an emulsion stabilizer.

TABLE IV

| Latex                | Protective Colloid                           | Resistance to Perchloroethylene |                |
|----------------------|--|---------------------------------|----------------|
|                      |  | Swelling (%)                    | Solubility (%) |
| Conventional product | surfactant                                   | 820                             | 28.5           |
| Example 7            | sodium polyacrylate                          | 310                             | 3.1            |
| do 8                 | sodium acrylate-methyl acrylate copolymer    | 110                             | 2.5            |
| do 9                 | methylolacrylamide-methyl acrylate copolymer | 39                              | 0.2            |
| do 10                | acrylamide-methyl methacrylate copolymer     | 52                              | 0.8            |

## EXAMPLE 11

5 A 5-necked 1-liter flask disposed in a hot water bath and equipped with a reflux condenser, a thermometer, a stirrer and a dropping funnel was charged with 305 grams of 3.5% aqueous solution of polyvinyl alcohol (degree of saponification 88.6%, degree of polymerization 1400) and 5 grams of isobutyl alcohol. While stirring, the temperature of the charge was raised to 75°C., after which one gram of potassium persulfate was added thereto and, at the same time, the addition dropwise of 190 grams of methyl methacrylate was begun from the dropping funnel. Thereafter, while continuing the dropping of the monomer over a period of 2 hours and 30 minutes  $\pm$  10 minutes at a rate as near constant as possible, the temperature of the hot water bath was so adjusted that a reaction temperature of 80—85°C. was maintained. After addition of monomer was complete, the temperature was maintained at 80—85°C. for another hour. The system was then cooled to below 30°C. and the latex was removed from the flask. This latex had a pH of 2.5 and it was very stable. The conversion was 98.7%.

## EXAMPLES 12—19

A 5-necked 1-liter flask disposed in a hot water bath and equipped with a reflux condenser, a thermometer, a stirrer and a dropping funnel was charged with a water-soluble high-molecular-weight substance indicated in Table V and also an emulsion stabilizer likewise indicated therein. Then, while stirring the liquid charged, its temperature was raised to 75°C., after which a catalyst, indicated in Table V, was added and, at the same time, the addition dropwise from the dropping funnel of a monomer, indicated in said Table V, was begun. Thereafter, while continuing the dropping of the monomer over a period of 2 hours and 30 minutes  $\pm$  10 minutes at a rate as near constant as possible, the temperature of the hot water bath was so adjusted that a reaction temperature of 80—85°C. would be maintained. After addition of monomer was complete, the temperature was held at 80—85°C. for another hour. The system was then cooled to below 30°C. and the emulsion was removed from the flask. The conversion and the pH of the resulting emulsions are shown in Table V.

TABLE V

| Example | Water-Soluble High Molecular Weight Substance  | Emulsion Stabilizer                      | Catalyst                                       | Monomer  | Conversion (%) | pH  |
|---------|--|--|--|--|----------------|-----|
| 12      | aq sol of 10 g ammonium polyacrylate in 290 g water  | octyl alcohol 12 g                       | aq sol of 1 g ammonium persulfate in 7 g water | ethyl acrylate 190 g                                     | 99.3           | 2.5 |
| 13      | 50 g 20% aq polyacrylamide copolymer sol diluted with 240 g water  | methyl isobutyl carbinol 6 g             | do   | mixture of 170 g butyl acrylate and 20 g n-butyl maleate | 99.3           | 2.5 |
| 14      | 4% aq polyvinyl alcohol sol (completely saponified product, D.P. 1700) 305 g   | 1,4-butanediol 10 g                      | do   | methyl acrylate 190 g                                    | 99.2           | 2.5 |
| 15      | 3% aq hydroxyethyl cellulose sol 305 g   | ethyl alcohol 12 g                       | potassium persulfate 1 g                       | ethyl acrylate 190 g                                     | 99.0           | 2.4 |
| 16      | 3.5% aq water-soluble starch sol 305 g   | isopropyl alcohol 8 g                    | aq sol of 1 g ammonium persulfate in 7 g water | do   | 99.5           | 2.4 |
| 17      | 3.5% aq polyvinyl alcohol sol (completely saponified product, D.P. 1000) 305 g   | isobutyl alcohol 3 g, 1,4-butanediol 5 g | potassium persulfate 1 g                       | do   | 99.6           | 2.5 |
| 18      | mixture of a sol consisting of 150 g of 2.8% ammonia water in which 5 g of casein was dissolved and 150 g 3% aq polyvinyl alcohol sol (completely saponified product, D.P. 1000) | isobutyl alcohol 6 g                     | aq sol of 1 g ammonium persulfate in 7 g water | mixture of 170 g ethyl acrylate and 20 g n-butyl maleate | 99.1           | 4.6 |
| 19      | 3.28% aq polyvinyl alcohol sol (completely saponified product, D.P. 1000) 305 g  | n-butyl alcohol 1 g                      | potassium persulfate 0.8 g                     | mixture of 180 g ethyl acrylate and 10 g vinyl acetate   | 99.2           | 2.5 |

## EXAMPLE 20

5 A 5-necked flask disposed in a hot water bath and equipped with a reflux condenser, a thermometer, a stirrer and a dropping funnel was charged with 286 grams of 3.5% aqueous solution of polyvinyl alcohol (degree of saponification 88.6%, average degree of polymerization 1000) and 10 grams of secondary butyl alcohol. The inside of the flask was purged with nitrogen and then, while stirring the solution and maintaining its temperature at 40°C., one gram of 30% aqueous hydrogen peroxide solution was added and, at the same time, the addition dropwise of a monomeric mixture consisting of 20 grams of acrylonitrile, 170 grams of n-butyl acrylate and 0.2 gram of methacrylic acid, as well as 20 grams of aqueous 5% solution of sodium formaldehyde-sulfoxylate was begun. The monomer and the aqueous solution were dropped over a period of 2 hours and 15 minutes  $\pm$  10 minutes at a rate as near constant as possible. Although a rise in temperature was noted upon initiation of the polymerization reaction, the temperature of the solution was maintained at 40  $\pm$  5°C. by cooling. After addition of monomer was complete, the polymerization was continued for a further one hour at this same temperature, after which the system was cooled. The resulting latex had a pH of 5.2 and the conversion was 99.6%.

## EXAMPLE 21

Example 20 was repeated except that 15 grams of polyoxyethylene lauryl ether were also added to the solution charged therein. The pH of the resulting latex was 5.2 and the conversion was 99.8%.

## EXAMPLE 22

The procedure of Example 15 was repeated except that 3 grams of isobutyl alcohol were used instead of 12 grams of ethyl alcohol as the emulsion stabilizer and a mixture comprising 190 grams of ethyl acrylate and 7 grams of isobutyl alcohol was charged dropwise instead merely of 190 grams of ethyl acrylate. The pH of the resulting latex was 2.4 and the conversion was 99.2%.

## EXAMPLES 23—30

The experiments were operated exactly as in Examples 12—19 except that the monomer dropping time was 2 hours 15 minutes  $\pm$  10 minutes. The class of the water-soluble high-molecular-weight substances, emulsion stabilizers, catalysts and monomers used in the several examples as well as the pH of the resulting latices and conversion are shown in Table VI.

TABLE VI

| Example | Water-Soluble High Molecular Weight Substance  | Emulsion Stabilizer                    | Catalyst                                       | Monomer  | Conversion (%) | pH  |
|---------|--|--|--|--|----------------|-----|
| 23      | 50 g 20% aq polyacrylamide copolymer sol dil with 240 g water  | allyl bromide 6 g                      | aq sol of 1 g ammonium persulfate in 7 g water | mixture of 170 g n-butyl acrylate and 20 g fumaric acid  | 98.3           | 2.5 |
| 24      | aq sol of 10 g ammonium polyacrylate in 290 g water  | allyl alcohol 5 g                      | do   | mixture of 171 g ethyl acrylate and 19 g n-butyl maleate | 99.5           | 2.5 |
| 25      | 2.8% aq hydroxyethyl cellulose solution 305 g  | do                                     | potassium persulfate 1 g                       | ethyl acrylate 190 g                                     | 99.9           | 2.5 |
| 26      | 3.31% aq water-soluble starch sol 305 g  | allyl acetate 10 g                     | potassium persulfate 0.8 g                     | do   | 98.8           | 2.8 |
| 27      | 3.28% aq polyvinyl alcohol sol (completely saponified product, D.P. 1000) 305 g  | allyl alcohol 15 g                     | aq sol of 1 g ammonium persulfate in 7 g water | methyl methacrylate 155 g                                | 97.4           | 2.5 |
| 28      | do   | allyl acetate 5 g<br>allyl alcohol 5 g | potassium persulfate 1 g                       | ethyl acrylate 190 g                                     | 99.7           | 2.5 |
| 29      | aq sol consisting of 295 g 1.4% ammonia water in which have been dissolved 5 g casein and 4.5 g polyvinyl alcohol (completely saponified product, D.P. 1000) | allyl alcohol 5 g                      | aq sol of 1 g ammonium persulfate in 7 g water | mixture of 171 g ethyl acrylate and 19 g n-butyl maleate | 99.0           | 4.5 |
| 30      | 3.28% aq polyvinyl alcohol sol (completely saponified product, D.P. 1000)  | allyl acetate 10 g                     | potassium persulfate 0.8 g                     | ethyl acrylate 180 g and vinyl propionate 10 g           | 98.9           | 2.5 |



**EXAMPLE 31**

Example 20 was repeated except that 1.5 grams of polyoxyethylene lauryl ether was added additionally to the liquid charged. The pH of the resulting latex was 6.0 and the conversion was 99.8%.

**COMPARATIVE EXAMPLE**

Examples 11—31 were repeated under exactly identical conditions excepting that the emulsion stabilizers were not used. In all cases, the state of emulsion was disrupted within one hour after the initiation of the dropping of the monomers, thus rendering it impossible to obtain stable latices.

**WHAT WE CLAIM IS:—**

1. A method of preparing an acrylic polymer latex by the aqueous emulsion polymerization of an acrylic and/or methacrylic ester, and optionally therewith a minor quantity of a comonomer, in the presence of a water-soluble high-molecular-weight substance as a protective colloid, which comprises adding to the polymerization system as an emulsion stabilizer 0.01—10 percent by weight, based on the total monomer, of allyl alcohol, an ester of allyl alcohol with an alkanolic acid an allyl halide or a saturated aliphatic alcohol of 2—8 carbon atoms.

2. A method according to claim 1 wherein 0.05—5% by weight of emulsion stabilizer, based on the total monomer, is added to the polymerization system.

3. A method according to claim 1 or 2

wherein the emulsion stabilizer is allyl alcohol, allyl acetate, allyl chloride, allyl bromide, ethyl alcohol, propyl alcohol, butyl alcohol, octyl alcohol, ethylene glycol, propylene glycol, butanediol, 1,8-octanediol or methylisobutylcarbinol.

4. A method according to any one of the preceding claims wherein the monomer polymerized is a methyl, ethyl, butyl, 2-ethylhexyl, glycidyl or hydroxyethyl ester of acrylic and/or methacrylic acid.

5. A method according to claim 4 wherein there is added to the acrylic and/or methacrylic ester as comonomer up to 5% by weight based on the total monomers of one or more of acrylic acid, methacrylic acid or vinyl sulphonic acid or their water soluble salts; up to 30% by weight based on the total monomers of at least one ester of maleic or fumaric acid; up to 30% by weight based on the total monomers of acrylonitrile and/or styrene; or up to 25% by weight based on the total monomers of vinyl acetate and/or vinyl propionate.

6. A method of preparing an acrylic polymer latex according to claim 1 substantially as hereinbefore described.

7. Acrylic polymer latices obtained by a method according to any one of the preceding claims.

J. A. KEMP & CO.,  
Chartered Patent Agents,  
14 South Square,  
Gray's Inn, London, W.C.1.

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COMPLETE SPECIFICATION

1 SHEET

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